A Stable Four-Membered N-Heterocyclic Carbene

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SUPPORTING INFORMATION

1. Synthesis and Spectroscopic Data

All manipulations were performed under an inert atmosphere of argon using standard Schlenk techniques. Dry, oxygen-free solvents were employed. ^{1}H, ^{13}C, ^{31}P, and ^{19}F NMR spectra were recorded on either a Varian Mercury-300, Varian Inova-500 or Varian Inova-600 NMR spectrometer. ^{1}H and ^{13}C NMR chemicals shifts are reported in ppm relative to Me_4Si as an external standard. ^{31}P and ^{19}F NMR chemical shifts are given relative to external standards of H_3PO_4 and $CFCl_3$, respectively.

Synthesis of the iminium salt **2a**: Aminodichlorophosphine (0.25 g, 1.4 mmol) followed by trimethylsilyl trifluoromethanesulfonate (0.32 g, 1.4 mmol) were added at room temperature to a dichloromethane solution of silylamidine (0.5 g, 1.4 mmol). After 30 minutes, the solvent was removed under vacuum and the crude residue was washed with ether, affording a white powder (0.5 g, 66%). Single crystals were obtained from a dichloromethane-toluene solution at -20 °C.
mp 133 °C dec; $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 300 K): d 126.9 (s); ^{19}F NMR (CD_2Cl_2, 300 K): d -79.6 (s, $CF_3SO_3^-$); ^{1}H NMR (CD_2Cl_2, 300 K): d 0.98 (br., 3H, CH_3), 1.24 (br., 3H, CH_3), 2.34 (s, 6H, CH_3), 2.44 (s, 12H, CH_3), 3.37 (br.; 4H, CH_2), 7.05 (s, 4H, H_{arom}), 9.09 (d, 1H, $^{3}J_{HP}$ = 2.7 Hz, NCHN); $^{13}C\{^{1}H\}$ NMR (CD_2Cl_2, 300 K)): d 13.5 (br., CH_3), 15.5 (br., CH_3), 19.2 (d, J_{CP} = 6 Hz, CH_3), 20.9 (s, CH_3), 41.8 (br., CH_2), 44.4 (br., CH_2), 120.9 (q, $^{1}J_{CF}$ = 319 Hz, CF_3), 130.3 (s, C_{arom}), 130.5 (s, CH_{arom}), 133.8 (s, C_{arom}), 139.8 (s, C_{arom}), 164.8 (d, $^{2}J_{CP}$ = 16 Hz, NCN).

Synthesis of the iminium salt **2b**: As described for **2a**, a white powder was obtained (90%). mp 163 °C dec; $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 300 K): d 135.0 (s); ^{19}F NMR (CD_2Cl_2, 300 K): d -79.6 (s, $CF_3SO_3^-$); ^{1}H NMR (CD_2Cl_2, 300 K): d 1.11 (d, 6H, $^{3}J_{HH}$ = 6.6 Hz, $CH_{3i\text{-}Pr}$), 1.41 (d, 6H, $^{3}J_{HH}$ = 7.2 Hz, $CH_{3i\text{-}Pr}$), 2.33 (s, 6H, CH_{3Mes}), 2.47 (s, 12H, CH_{3Mes}), 3.64 (m, 1H, CH), 3.91 (m, 1H, CH), 7.04 (s, 4H, H_{arom}), 8.97 (d, 1H, $^{3}J_{HP}$ = 2.2 Hz, NCHN); $^{13}C\{^{1}H\}$ NMR (CD_2Cl_2, 300 K): d 19.1 (d, J_{CP} = 7 Hz, CH_3), 20.9 (s, CH_3), 21.4 (s, CH_3), 27.2 (d, J_{CP} = 13 Hz, CH_3), 47.7 (d, $^{2}J_{CP}$ = 30 Hz, CH), 51.6 (d, $^{2}J_{CP}$ = 9 Hz, CH), 120.9 (q, $^{1}J_{CF}$ = 319 Hz, CF_3), 130.1 (s, C_{arom}), 130.6 (s, CH_{arom}), 133.3 (s, C_{arom}), 139.6 (s, C_{arom}), 164.2 (d, $^{2}J_{CP}$ = 17 Hz, NCHN).

Synthesis of the iminium salt **2c**: As described for **2a**, white powder (96%). mp 154 °C dec; $^{31}P\{^{1}H\}$ NMR (CD_2Cl_2, 300 K): d 136.5 (s); ^{19}F NMR (CD_2Cl_2, 300 K): -79.6 (s, $CF_3SO_3^-$), ^{1}H NMR (CD_2Cl_2, 300 K): d 1.15 (d, 6H, $^{3}J_{HH}$ = 6.3 Hz, $CH_{3Ni\text{-}Pr}$), 1.35 (d, 24H, $^{3}J_{HH}$ = 6.6 Hz, CH_{3Ar}), 1.42 (d, 6H, $^{3}J_{HH}$ = 6.6 Hz, $CH_{3Ni\text{-}Pr}$), 3.20 (sept.d, 4H, $^{3}J_{HH}$ = 6.6 Hz, $^{5}J_{HP}$ = 2.7 Hz, CH), 3.68 (m, 1H, CH), 3.89 (m, 1H, $CH_{i\text{-}Pr}$), 7.36 (d, 4H, $^{3}J_{HH}$ = 7.5 Hz, H_{arom}), 7.49 (t, 2H, $^{3}J_{HH}$ = 7.5 Hz, H_{arom}), 8.65 (d, 1H, $^{3}J_{HP}$ = 2.1 Hz, NCHN); $^{13}C\{^{1}H\}$ NMR (CD_2Cl_2, 300 K): d 22.0 (s, CH_3), 24.4 (br., CH_3), 27.4 (d, J_{CP} = 13 Hz, CH_3), 29.9 (d, J_{CP} = 7 Hz, CH), 48.0 (d, $^{2}J_{CP}$ = 28 Hz, CH), 120.9 (q, $^{1}J_{CF}$ = 319 Hz, CF_3), 125.1 (s, CH_{arom}), 129.4 (d, J_{CP} = 2 Hz, C_{arom}), 130.6 (s, CH_{arom}), 144.8 (s, C_{arom}), 163.0 (d, $^{2}J_{CP}$ = 17 Hz, NCHN).

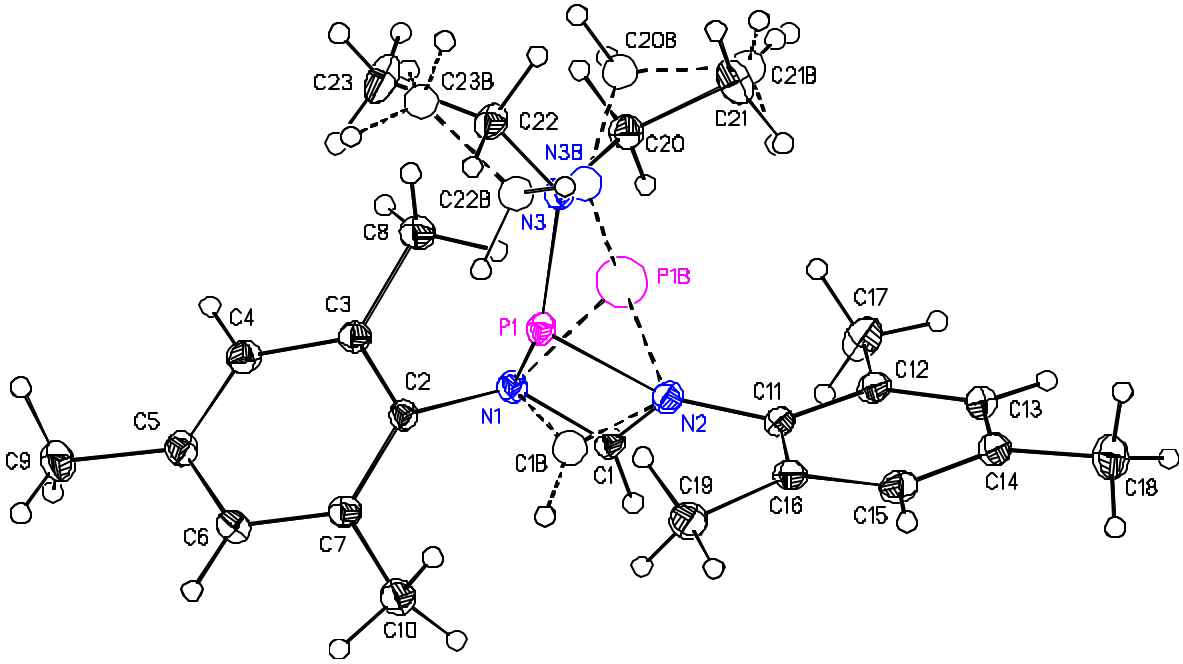
Reaction of the iminium salt **2b** *with potassium tert-butoxide* (**3**): Potassium *tert*-butoxide (0.012 g, 0.1 mmol) was added at room temperature to a toluene solution of iminium salt **2b** (0.06 g, 0.1 mmol). After 30 minutes, the solution was filtered and evaporated under vacuum, affording a yellow powder (0.041 g, 86%). mp 165 °C; $^{31}P\{^{1}H\}$ NMR (C_6D_6, 300 K): d 124.6 (s); ^{1}H NMR

(C_6D_6, 300 K): d 0.90 (s, 9H, $CH_{3t\text{-Bu}}$), 0.96 (br., 6H, CH_3), 1.09 (br., 6H, CH_3), 2.14 (s, 3H, CH_3), 2.15 (s, 3H, CH_3), 2.28 (s, 6H, CH_3), 2.55 (s, 3H, CH_3), 2.57 (s, 3H, CH_3), 4.73 (br., 2H, CH), 6.83 (s, 2H, H_{arom}), 6.84 (s, 1H, H_{arom}), 6.88 (s, 1H, H_{arom}), 7.87 (d, 1H, $^3J_{HP}$ = 3.6 Hz, NCHN); $^{13}C\{^1H\}$ NMR (C_6D_6, 300 K): d 19.3 (s, CH_3), 19.5 (s, CH_3), 19.8 (s, CH_3), 20.8 (s, CH_3), 20.9 (s, CH_3), 24.3 (br., CH_3), 29.9 (d, J_{CP} = 8 Hz, CH_3), 44.8 (br., CH), 76.6 (d, $^2J_{CP}$ = 15 Hz, $C_{t\text{-Bu}}$), 128.5 (s), 128.9 (s), 129.2 (s), 130.8 (s), 136.0 (s), 136.5 (s), 139.5 (s), 149.5 (s), 150.9 (d, $^2J_{CP}$ = 10 Hz, NCHN).

Reaction of the iminium salt **2b** *with mesityllithium or potassium hexamethyldisilazide* (**4**)*:* Mesityllithium (0.092 g, 0.73 mmol) or potassium hexamethyldisilazide (0.14 g, 0.73 mmol) was added at room temperature to a toluene solution of iminium salt **2b** (0.37 g, 0.66 mmol). After one hour, the solution was filtered and evaporated under vacuum, affording a yellow powder (0.24 g, 45%). Single crystals were obtained by slow evaporation of a pentane solution at room temperature. mp 174 °C; $^{31}P\{^1H\}$ NMR (C_6D_6, 300 K): d 155.7 (s); ^1H NMR (C_6D_6, 300 K): d 0.82 (d, 12H, $^3J_{HH}$ = 6.0 Hz, CH_3), 1.09 (d, 12H, $^3J_{HH}$ = 6.5 Hz, CH_3), 2.10 (s, 12H, CH_3), 2.29 (s, 12H, CH_3), 2.92 (s, 12H, CH_3), 4.70 (m, 4H, CH), 6.32 (s, 4H, H_{arom}), 6.66 (s, 4H, H_{arom}); $^{13}C\{^1H\}$ NMR (C_6D_6, 300 K): d 20.7 (s, CH_3), 21.1 (d, J_{CP} = 9 Hz, CH_3), 21.5 (d, J_{CP} = 16 Hz, CH_3), 21.7 (d, J_{CP} = 17 Hz, CH_3), 21.9 (d, J_{CP} = 7 Hz, CH_3), 27.0 (d, J_{CP} = 13 Hz, CH_3), 27.1 (d, J_{CP} = 14 Hz, CH_3), 44.5 (d, $^2J_{CP}$ = 9 Hz, CH), 44.7 (d, $^2J_{CP}$ = 5 Hz, CH), 126.5 (d, J_{CP} = 8 Hz, NCN), 129.1 (d, J_{CP} = 23 Hz), 129.7 (d, J_{CP} = 6 Hz), 132.9 (d, J_{CP} = 2 Hz), 134.5 (s), 136.2 (s), 137.5 (d, J_{CP} = 5 Hz), C_{arom} under the C_6D_6 peak.

Reaction of the iminium salt **2c** *with mesityllithium or potassium hexamethyldisilazide* (**5**): Mesityllithium (0.011 g, 0.086 mmol) or potassium hexamethyldisilazide (0.017 g, 0.086 mmol) was added at room temperature to a toluene solution of iminium salt **2c** (0.05 g, 0.078 mmol). After 10 minutes, pentane was added and the solution was filtered. The solvents were removed under vacuum affording a white powder (0.022 g, 47%). Single crystals were obtained from a saturated pentane solution at -50 °C. mp: the crystals were not stable 2 hours at room temperature; $^{31}P\{^{1}H\}$ NMR (C_7D_8, 278 K): d 157.2 (s); ^{1}H NMR (C_7D_8, 278 K): d 1.39 (d, 6H, J_{CP} = 8.2 Hz, CH_3), 1.05 (d, 6H, J_{CP} = 9.0 Hz, CH_3), 1.32 (d, 3H, J_{CP} = 8.4 Hz, CH_3), 1.46 (d, 12H, J_{CP} = 8.4 Hz, CH_3), 1.54 (d, 9H, J_{CP} = 8.5 Hz, CH_3), 2.87 (m, 1H, CH), 4.04 (m, 4H, CH), 4.24 (m, 1H, CH), 7.00-7.10 (m, 6H, H_{arom}); $^{13}C\{^{1}H\}$ NMR (C_7D_8, 278 K): d 22.1 (s, CH_3), 23.8 (br., CH_3), 25.50 (br., CH_3), 26.8 (d, J_{CP} = 13 Hz, CH_3), 29.1 (d, J_{CP} = 8 Hz, CH), 44.6 (d, $^{2}J_{CP}$ = 27 Hz, CH), 48.7 (d, $^{2}J_{CP}$ = 8 Hz, CH), 123.6 (s), 125.2 (s), 126.9 (s), 128.1 (s), 129.0 (s), 137,1 (s), 145.5 (s), 285.0 (d, $^{2}J_{CP}$ = 13 Hz, NCN).

2. X-Ray Crystallographic Data



2a

Special Refinement Details

The crystals contain two different conformationaly distinct compounds. The two conformations differ by what might be called an inversion of the tetrahedral geometry around phosphorous but both share N1, N2 and the aromatic constituents.. Although one conformation comprises only 5% of the structure (see Table 2 and Figure 2) it is clear in the electron density maps and is included in the model with restraints. These include one common isotropic temperature factor, bond distance and angle restraints and planarity restraints for C1B, N1, P1B and N2.

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 1. Crystal data and structure refinement for 2a.

Identification code	**2a**	
Empirical formula	$[C_{23}H_{33}N_3P]^+[SO_3CF_3]?$	
Formula weight	531.56	
Crystal size	0.37 x 0.26 x 0.25 mm^3	
Crystal color	Colorless	
Wavelength	0.71073 Å MoKα	
Data Collection Temperature	98(2) K	
Unit cell dimensions	a = 10.5668(3) Å b = 11.8758(4) Å c = 12.7952(4) Å	α= 67.7490(10)° β= 65.7880(10)° γ = 67.4890(10)°
Volume	1305.19(7) $Å^3$	
Z	2	
Crystal system	Triclinic	
Space group	P-1	
Density (calculated)	1.353 Mg/m^3	
F(000)	560	
θ range for data collection	1.81 to 41.76°	
Completeness to θ = 41.76°	83.8 %	
Index ranges	$-19 \le h \le 19, -20 \le k \le 21, -22 \le l \le 22$	
Reflections collected	50465	
Independent reflections	15049 [R_{int}= 0.0566]	
Absorption coefficient	0.237 mm^{-1}	
Absorption correction	None	
Max. and min. transmission	0.9430 and 0.9173	
Refinement method	Full matrix least-squares on F^2	
Data / restraints / parameters	15049 / 9 / 348	
Goodness-of-fit on F^2	1.703	
Final R indices [I>2σ(I), 10187 reflections]	R1 = 0.0450, *w*R2 = 0.0829	
R indices (all data)	R1 = 0.0719, *w*R2 = 0.0850	
Largest diff. peak and hole	0.996 and -0.805 e.$Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 2a. U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}	Occ
S(1)	7789(1)	7832(1)	2538(1)	16(1)	1
F(1)	7076(1)	6400(1)	1849(1)	25(1)	1
F(2)	6513(1)	6011(1)	3751(1)	27(1)	1
F(3)	5261(1)	7674(1)	2784(1)	29(1)	1
O(1)	9138(1)	6886(1)	2542(1)	30(1)	1
O(2)	7761(1)	8765(1)	1422(1)	25(1)	1
O(3)	7078(1)	8349(1)	3559(1)	22(1)	1
C(24)	6596(1)	6939(1)	2739(1)	17(1)	1
P(1)	4533(1)	3200(1)	1785(1)	13(1)	0.947(1)
N(1)	5943(1)	2276(1)	2484(1)	14(1)	1
N(2)	4395(1)	1589(1)	2468(1)	15(1)	1
N(3)	3245(1)	4079(1)	2612(1)	14(1)	0.947(1)
C(1)	5660(1)	1210(1)	2653(1)	13(1)	0.947(1)
C(2)	7281(1)	2538(1)	2228(1)	13(1)	1
C(3)	7346(1)	3094(1)	2990(1)	15(1)	1
C(4)	8567(1)	3500(1)	2654(1)	17(1)	1
C(5)	9664(1)	3399(1)	1591(1)	16(1)	1
C(6)	9581(1)	2809(1)	874(1)	17(1)	1
C(7)	8395(1)	2366(1)	1174(1)	15(1)	1
C(8)	6130(1)	3303(1)	4104(1)	20(1)	1
C(9)	10906(1)	3953(1)	1216(1)	23(1)	1
C(10)	8339(1)	1746(1)	365(1)	19(1)	1
C(11)	3517(1)	877(1)	2530(1)	14(1)	1
C(12)	2778(1)	217(1)	3645(1)	15(1)	1
C(13)	1830(1)	-365(1)	3677(1)	18(1)	1
C(14)	1611(1)	-297(1)	2651(1)	18(1)	1
C(15)	2420(1)	318(1)	1558(1)	18(1)	1
C(16)	3387(1)	901(1)	1474(1)	15(1)	1
C(17)	3013(1)	110(1)	4764(1)	23(1)	1
C(18)	507(1)	-859(1)	2718(1)	28(1)	1
C(19)	4284(1)	1517(1)	272(1)	22(1)	1
C(20)	2605(1)	3697(1)	3917(1)	17(1)	0.947(1)
C(21)	1074(2)	3603(2)	4277(1)	24(1)	0.947(1)
C(22)	2776(1)	5447(1)	2015(1)	17(1)	0.947(1)
C(23)	3530(1)	6227(1)	2158(1)	27(1)	0.947(1)
P(1B)	4263(5)	2270(5)	3669(5)	26(1)	0.053(1)
N(3B)	3251(17)	3703(15)	3252(14)	26(1)	0.053(1)
C(1B)	5674(9)	1778(5)	1872(8)	26(1)	0.053(1)
C(20B)	2138(18)	4190(20)	4196(16)	26(1)	0.053(1)
C(21B)	950(30)	3530(40)	4590(30)	26(1)	0.053(1)
C(22B)	3400(20)	4547(15)	2099(16)	26(1)	0.053(1)
C(23B)	3320(20)	5898(17)	2060(20)	26(1)	0.053(1)

Table 3. Bond lengths [Å] and angles [°] for 2a

S(1)-O(1)	1.4393(7)	F(3)-C(24)-F(1)	107.72(7)
S(1)-O(2)	1.4422(7)	F(3)-C(24)-F(2)	107.67(7)
S(1)-O(3)	1.4478(7)	F(1)-C(24)-F(2)	107.37(7)
S(1)-C(24)	1.8259(9)	F(3)-C(24)-S(1)	111.68(6)
F(1)-C(24)	1.3386(10)	F(1)-C(24)-S(1)	111.33(6)
F(2)-C(24)	1.3414(11)	F(2)-C(24)-S(1)	110.88(6)
F(3)-C(24)	1.3358(10)	N(3)-P(1)-N(2)	107.97(4)
P(1)-N(3)	1.6219(8)	N(3)-P(1)-N(1)	109.67(4)
P(1)-N(2)	1.8136(8)	N(2)-P(1)-N(1)	70.49(3)
P(1)-N(1)	1.8237(8)	C(1B)-N(1)-C(1)	43.1(3)
N(1)-C(1B)	1.307(9)	C(1B)-N(1)-C(2)	126.4(4)
N(1)-C(1)	1.3306(11)	C(1)-N(1)-C(2)	131.05(7)
N(1)-C(2)	1.4427(10)	C(1B)-N(1)-P(1B)	95.4(5)
N(1)-P(1B)	1.801(5)	C(1)-N(1)-P(1B)	74.02(17)
N(2)-C(1B)	1.310(9)	C(2)-N(1)-P(1B)	137.88(17)
N(2)-C(1)	1.3279(11)	C(1B)-N(1)-P(1)	61.1(2)
N(2)-C(11)	1.4395(10)	C(1)-N(1)-P(1)	91.02(5)
N(2)-P(1B)	1.927(5)	C(2)-N(1)-P(1)	127.89(6)
N(3)-C(20)	1.4780(13)	P(1B)-N(1)-P(1)	74.17(17)
N(3)-C(22)	1.4903(12)	C(1B)-N(2)-C(1)	43.1(3)
C(2)-C(3)	1.4028(11)	C(1B)-N(2)-C(11)	139.6(4)
C(2)-C(7)	1.4031(11)	C(1)-N(2)-C(11)	130.79(7)
C(3)-C(4)	1.3952(12)	C(1B)-N(2)-P(1)	61.4(2)
C(3)-C(8)	1.5052(12)	C(1)-N(2)-P(1)	91.55(5)
C(4)-C(5)	1.3897(12)	C(11)-N(2)-P(1)	135.89(6)
C(5)-C(6)	1.3940(12)	C(1B)-N(2)-P(1B)	89.6(4)
C(5)-C(9)	1.5127(12)	C(1)-N(2)-P(1B)	69.62(16)
C(6)-C(7)	1.3971(12)	C(11)-N(2)-P(1B)	128.67(16)
C(7)-C(10)	1.5083(12)	P(1)-N(2)-P(1B)	71.43(16)
C(11)-C(16)	1.4010(12)	C(20)-N(3)-C(22)	116.49(7)
C(11)-C(12)	1.4033(12)	C(20)-N(3)-P(1)	126.69(7)
C(12)-C(13)	1.3985(12)	C(22)-N(3)-P(1)	116.43(7)
C(12)-C(17)	1.5033(12)	N(2)-C(1)-N(1)	104.29(7)
C(13)-C(14)	1.3921(13)	C(3)-C(2)-C(7)	122.50(7)
C(14)-C(15)	1.3941(13)	C(3)-C(2)-N(1)	117.55(7)
C(14)-C(18)	1.5112(12)	C(7)-C(2)-N(1)	119.55(7)
C(15)-C(16)	1.3897(12)	C(4)-C(3)-C(2)	117.41(8)
C(16)-C(19)	1.5112(12)	C(4)-C(3)-C(8)	120.43(8)
C(20)-C(21)	1.5267(15)	C(2)-C(3)-C(8)	122.11(7)
C(22)-C(23)	1.5305(13)	C(5)-C(4)-C(3)	121.91(8)
P(1B)-N(3B)	1.647(17)	C(4)-C(5)-C(6)	118.95(8)
N(3B)-C(22B)	1.420(9)	C(4)-C(5)-C(9)	119.99(8)
N(3B)-C(20B)	1.421(9)	C(6)-C(5)-C(9)	121.04(8)
C(20B)-C(21B)	1.546(10)	C(5)-C(6)-C(7)	121.60(8)
C(22B)-C(23B)	1.556(10)	C(6)-C(7)-C(2)	117.53(8)
		C(6)-C(7)-C(10)	119.91(8)
O(1)-S(1)-O(2)	115.76(5)	C(2)-C(7)-C(10)	122.56(7)
O(1)-S(1)-O(3)	115.25(4)	C(16)-C(11)-C(12)	121.97(7)
O(2)-S(1)-O(3)	114.03(4)	C(16)-C(11)-N(2)	118.88(7)
O(1)-S(1)-C(24)	103.24(4)	C(12)-C(11)-N(2)	119.11(7)
O(2)-S(1)-C(24)	103.38(4)	C(13)-C(12)-C(11)	117.44(8)
O(3)-S(1)-C(24)	102.70(4)	C(13)-C(12)-C(17)	120.95(8)

C(11)-C(12)-C(17)	121.60(8)	N(3)-C(22)-C(23)	111.74(8)
C(14)-C(13)-C(12)	122.07(8)	N(3B)-P(1B)-N(1)	102.2(6)
C(13)-C(14)-C(15)	118.41(8)	N(3B)-P(1B)-N(2)	98.4(6)
C(13)-C(14)-C(18)	120.96(8)	N(1)-P(1B)-N(2)	68.43(18)
C(15)-C(14)-C(18)	120.62(8)	C(22B)-N(3B)-C(20B)	115.8(16)
C(16)-C(15)-C(14)	121.87(8)	C(22B)-N(3B)-P(1B)	129.2(13)
C(15)-C(16)-C(11)	118.05(8)	C(20B)-N(3B)-P(1B)	114.9(14)
C(15)-C(16)-C(19)	120.28(8)	N(2)-C(1B)-N(1)	106.7(7)
C(11)-C(16)-C(19)	121.66(8)	N(3B)-C(20B)-C(21B)	105.5(18)
N(3)-C(20)-C(21)	111.86(8)	N(3B)-C(22B)-C(23B)	113.1(16)

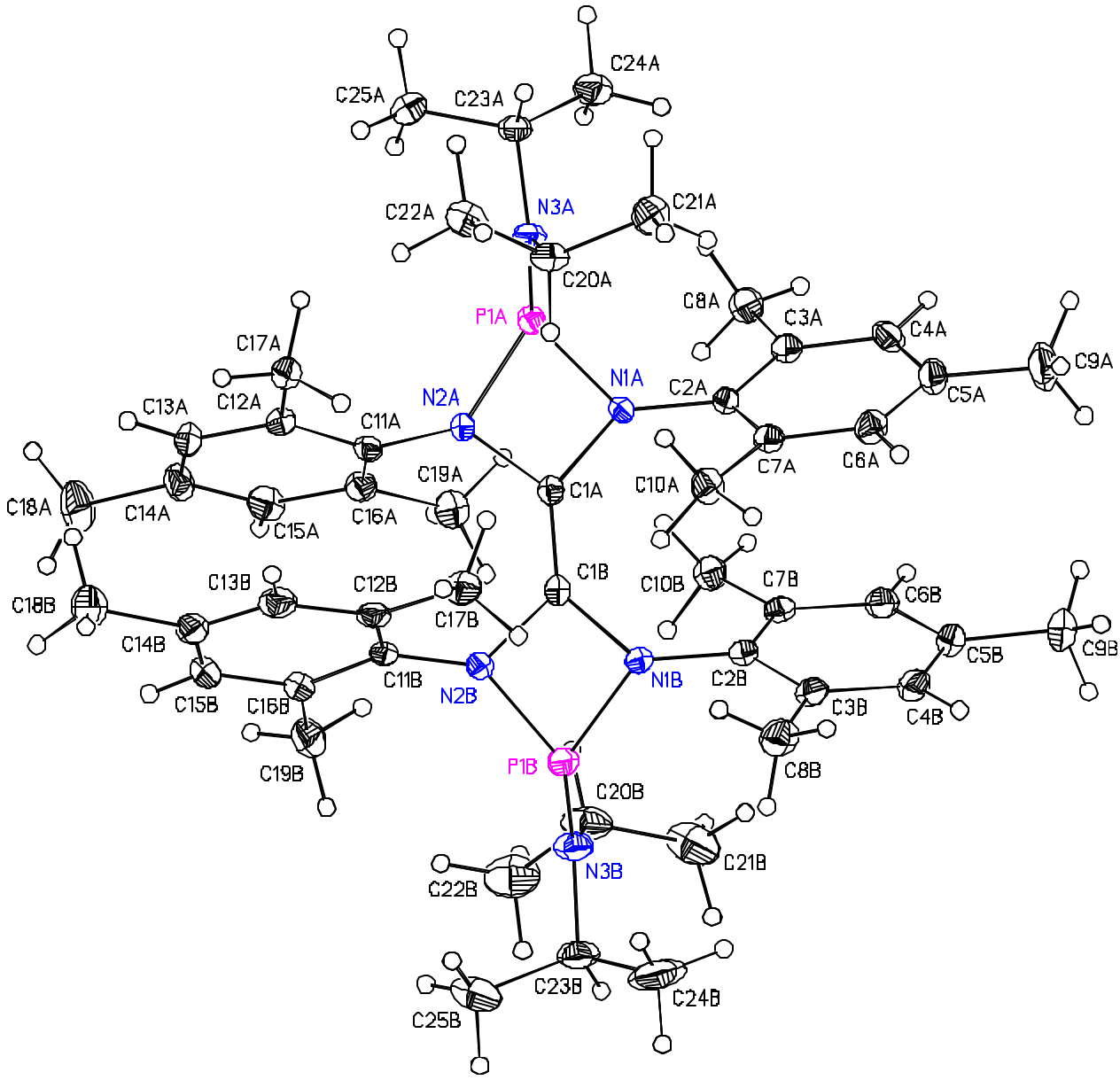
Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for 2a. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	164(1)	146(1)	180(1)	-50(1)	-55(1)	-42(1)
F(1)	343(3)	241(3)	243(3)	-129(2)	-91(2)	-83(2)
F(2)	347(3)	207(3)	203(3)	-27(2)	-11(2)	-136(2)
F(3)	156(3)	324(3)	408(4)	-149(3)	-81(3)	-34(2)
O(1)	165(3)	272(4)	455(5)	-140(3)	-115(3)	7(3)
O(2)	345(4)	219(4)	180(3)	-8(3)	-57(3)	-139(3)
O(3)	334(4)	173(3)	209(3)	-81(3)	-122(3)	-39(3)
C(24)	188(4)	161(4)	171(4)	-66(3)	-38(3)	-40(3)
P(1)	136(1)	114(1)	143(1)	-16(1)	-55(1)	-37(1)
N(1)	129(3)	138(3)	150(3)	-36(3)	-44(3)	-37(2)
N(2)	153(3)	139(3)	177(3)	-46(3)	-57(3)	-43(3)
N(3)	144(3)	106(4)	162(4)	-10(3)	-56(3)	-29(3)
C(1)	140(4)	101(4)	139(4)	-20(3)	-51(3)	-25(3)
C(2)	118(3)	118(4)	156(4)	-27(3)	-50(3)	-33(3)
C(3)	139(4)	133(4)	163(4)	-39(3)	-51(3)	-26(3)
C(4)	157(4)	169(4)	201(4)	-70(3)	-73(3)	-32(3)
C(5)	130(4)	161(4)	200(4)	-39(3)	-67(3)	-35(3)
C(6)	135(4)	186(4)	163(4)	-53(3)	-28(3)	-41(3)
C(7)	153(4)	134(4)	143(4)	-32(3)	-53(3)	-27(3)
C(8)	189(4)	227(5)	193(4)	-99(4)	-18(3)	-72(3)
C(9)	159(4)	279(5)	268(5)	-94(4)	-44(4)	-89(4)
C(10)	194(4)	243(5)	174(4)	-88(4)	-31(3)	-85(3)
C(11)	133(4)	124(4)	165(4)	-54(3)	-41(3)	-35(3)
C(12)	180(4)	134(4)	143(4)	-47(3)	-39(3)	-36(3)
C(13)	174(4)	161(4)	174(4)	-50(3)	-10(3)	-67(3)
C(14)	138(4)	174(4)	241(4)	-88(4)	-46(3)	-39(3)
C(15)	191(4)	185(4)	180(4)	-78(3)	-74(3)	-34(3)
C(16)	156(4)	140(4)	144(4)	-49(3)	-35(3)	-27(3)
C(17)	360(5)	201(5)	165(4)	-23(4)	-96(4)	-119(4)
C(18)	219(5)	331(6)	350(6)	-135(5)	-64(4)	-130(4)
C(19)	262(5)	227(5)	155(4)	-62(4)	-22(4)	-95(4)
C(20)	173(4)	164(5)	157(4)	-37(4)	-47(4)	-44(3)
C(21)	204(5)	304(6)	193(6)	-34(6)	-19(5)	-115(4)
C(22)	167(4)	113(4)	233(5)	-14(3)	-96(4)	-24(3)
C(23)	330(6)	173(5)	372(6)	-21(5)	-169(5)	-119(4)

Table 5. Torsion angles [°] for 2a.

C(3)-C(2)-N(1)-P(1)	100.02(9)
C(3)-C(2)-N(1)-P(1B)	-10.2(3)
C(12)-C(11)-N(2)-P(1)	-126.93(8)
C(12)-C(11)-N(1)-P(1B)	-37.8(2)



4

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 1. Crystal data and structure refinement for EMD05 (CCDC 231890).

Identification code	**4**
Empirical formula	$C_{50}H_{72}N_6P_2$
Formula weight	819.08
Crystal size	0.33 x 0.31 x 0.21 mm^3
Crystal color	Yellow
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
Unit cell dimensions	a = 13.7626(6) Å b = 16.2123(8) Å β= 101.3420(10)° c = 21.7085(10) Å
Volume	4749.1(4) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/c$
Density (calculated)	1.146 Mg/m^3
F(000)	1776
θ range for data collection	1.58 to 31.93°
Completeness to θ = 31.93°	88.9 %
Index ranges	$-20 \le h \le 20$, $-21 \le k \le 23$, $-28 \le l \le 31$
Reflections collected	76278
Independent reflections	14587 [R_{int} = 0.0787]
Absorption coefficient	0.131 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9730 and 0.9580
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	14587 / 0 / 811
Goodness-of-fit on F^2	1.445
Final R indices [$I>2\sigma(I)$, 8893 reflections]	R1 = 0.0500, *w*R2 = 0.0728
R indices (all data)	R1 = 0.0950, *w*R2 = 0.0770
Largest diff. peak and hole	0.698 and -0.521 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 4. U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
P(1A)	3675(1)	2692(1)	196(1)	15(1)
N(1A)	2622(1)	2659(1)	536(1)	15(1)
N(2A)	4045(1)	3153(1)	937(1)	14(1)
N(3A)	4103(1)	1732(1)	205(1)	16(1)
C(1A)	3045(1)	3175(1)	1058(1)	14(1)
C(2A)	1716(1)	2215(1)	366(1)	15(1)
C(3A)	1197(1)	2243(1)	-259(1)	17(1)
C(4A)	325(1)	1787(1)	-430(1)	22(1)
C(5A)	-63(1)	1319(1)	-3(1)	23(1)
C(6A)	468(1)	1298(1)	609(1)	21(1)
C(7A)	1358(1)	1716(1)	803(1)	17(1)
C(8A)	1532(1)	2762(1)	-755(1)	22(1)
C(9A)	-1024(1)	852(1)	-189(1)	35(1)
C(10A)	1919(1)	1582(1)	1462(1)	21(1)
C(11A)	4770(1)	3807(1)	1025(1)	16(1)
C(12A)	5750(1)	3584(1)	1296(1)	18(1)
C(13A)	6479(1)	4191(1)	1381(1)	21(1)
C(14A)	6274(1)	5005(1)	1207(1)	22(1)
C(15A)	5314(1)	5199(1)	914(1)	22(1)
C(16A)	4554(1)	4616(1)	812(1)	18(1)
C(17A)	6022(1)	2712(1)	1482(1)	21(1)
C(18A)	7066(1)	5658(1)	1340(1)	36(1)
C(19A)	3535(1)	4869(1)	476(1)	22(1)
C(20A)	4037(1)	1102(1)	689(1)	20(1)
C(21A)	3281(1)	437(1)	434(1)	26(1)
C(22A)	5036(1)	717(1)	968(1)	25(1)
C(23A)	4520(1)	1451(1)	-345(1)	19(1)
C(24A)	3789(1)	1517(1)	-963(1)	26(1)
C(25A)	5497(1)	1875(1)	-368(1)	30(1)
P(1B)	2181(1)	4122(1)	2386(1)	17(1)
N(1B)	1729(1)	3657(1)	1661(1)	15(1)
N(2B)	3183(1)	4125(1)	1992(1)	15(1)
N(3B)	1672(1)	5054(1)	2364(1)	23(1)
C(1B)	2681(1)	3602(1)	1488(1)	14(1)
C(2B)	751(1)	3461(1)	1340(1)	17(1)
C(3B)	121(1)	3016(1)	1659(1)	19(1)
C(4B)	-841(1)	2843(1)	1343(1)	24(1)
C(5B)	-1193(1)	3075(1)	727(1)	24(1)
C(6B)	-558(1)	3520(1)	427(1)	21(1)
C(7B)	397(1)	3731(1)	720(1)	17(1)
C(8B)	450(1)	2698(1)	2319(1)	27(1)
C(9B)	-2222(1)	2852(1)	383(1)	34(1)
C(10B)	1010(1)	4257(1)	373(1)	20(1)
C(11B)	4189(1)	4023(1)	2325(1)	16(1)
C(12B)	4567(1)	3262(1)	2570(1)	19(1)
C(13B)	5559(1)	3215(1)	2868(1)	24(1)

C(14B)	6180(1)	3900(1)	2933(1)	26(1)
C(15B)	5770(1)	4648(1)	2725(1)	25(1)
C(16B)	4782(1)	4731(1)	2426(1)	20(1)
C(17B)	3933(1)	2503(1)	2530(1)	22(1)
C(18B)	7272(1)	3811(2)	3215(1)	41(1)
C(19B)	4369(1)	5566(1)	2231(1)	28(1)
C(20B)	1435(1)	5589(1)	1803(1)	32(1)
C(21B)	328(2)	5615(1)	1542(1)	43(1)
C(22B)	1851(2)	6458(1)	1912(1)	40(1)
C(23B)	1350(1)	5357(1)	2943(1)	27(1)
C(24B)	691(2)	4739(1)	3194(1)	55(1)
C(25B)	2227(2)	5612(1)	3445(1)	35(1)

Table 3. Bond lengths [Å] and angles [°] for 4.

P(1A)-N(3A)	1.6626(11)	C(20A)-C(22A)	1.523(2)
P(1A)-N(1A)	1.7513(11)	C(20A)-C(21A)	1.526(2)
P(1A)-N(2A)	1.7557(11)	C(20A)-H(20A)	1.003(12)
P(1A)-C(1A)	2.3478(14)	C(21A)-H(21A)	0.978(14)
N(1A)-C(2A)	1.4239(15)	C(21A)-H(21B)	1.030(14)
N(1A)-C(1A)	1.4374(16)	C(21A)-H(21C)	0.994(15)
N(2A)-C(11A)	1.4436(16)	C(22A)-H(22A)	1.011(14)
N(2A)-C(1A)	1.4520(15)	C(22A)-H(22B)	1.008(15)
N(3A)-C(20A)	1.4798(17)	C(22A)-H(22C)	0.960(15)
N(3A)-C(23A)	1.4937(16)	C(23A)-C(24A)	1.515(2)
C(1A)-C(1B)	1.3335(17)	C(23A)-C(25A)	1.520(2)
C(2A)-C(3A)	1.4056(18)	C(23A)-H(23A)	0.982(12)
C(2A)-C(7A)	1.4085(18)	C(24A)-H(24A)	0.990(14)
C(3A)-C(4A)	1.3953(18)	C(24A)-H(24B)	0.999(14)
C(3A)-C(8A)	1.508(2)	C(24A)-H(24C)	0.962(15)
C(4A)-C(5A)	1.386(2)	C(25A)-H(25A)	0.998(17)
C(4A)-H(4A)	0.979(12)	C(25A)-H(25B)	0.971(15)
C(5A)-C(6A)	1.385(2)	C(25A)-H(25C)	1.020(15)
C(5A)-C(9A)	1.509(2)	P(1B)-N(3B)	1.6630(12)
C(6A)-C(7A)	1.3898(18)	P(1B)-N(1B)	1.7451(11)
C(6A)-H(6A)	0.979(13)	P(1B)-N(2B)	1.7595(11)
C(7A)-C(10A)	1.501(2)	P(1B)-C(1B)	2.3482(14)
C(8A)-H(8A)	0.967(15)	N(1B)-C(2B)	1.4237(16)
C(8A)-H(8B)	0.981(16)	N(1B)-C(1B)	1.4358(15)
C(8A)-H(8C)	0.969(14)	N(2B)-C(11B)	1.4387(15)
C(9A)-H(9A)	0.980(19)	N(2B)-C(1B)	1.4490(16)
C(9A)-H(9B)	0.974(19)	N(3B)-C(20B)	1.4797(19)
C(9A)-H(9C)	0.994(19)	N(3B)-C(23B)	1.4947(18)
C(10A)-H(10A)	1.003(14)	C(2B)-C(7B)	1.4082(18)
C(10A)-H(10B)	0.998(14)	C(2B)-C(3B)	1.4088(18)
C(10A)-H(10C)	0.956(15)	C(3B)-C(4B)	1.3947(19)
C(11A)-C(16A)	1.4021(18)	C(3B)-C(8B)	1.507(2)
C(11A)-C(12A)	1.4083(17)	C(4B)-C(5B)	1.382(2)
C(12A)-C(13A)	1.3909(18)	C(4B)-H(4B)	0.970(13)
C(12A)-C(17A)	1.4987(19)	C(5B)-C(6B)	1.388(2)
C(13A)-C(14A)	1.386(2)	C(5B)-C(9B)	1.509(2)
C(13A)-H(13A)	0.990(12)	C(6B)-C(7B)	1.3851(18)
C(14A)-C(15A)	1.3845(19)	C(6B)-H(6B)	0.953(12)
C(14A)-C(18A)	1.507(2)	C(7B)-C(10B)	1.5019(19)
C(15A)-C(16A)	1.3959(19)	C(8B)-H(8D)	0.938(19)
C(15A)-H(15A)	0.936(13)	C(8B)-H(8E)	0.961(16)
C(16A)-C(19A)	1.5043(19)	C(8B)-H(8F)	0.954(18)
C(17A)-H(17A)	1.002(14)	C(9B)-H(9D)	0.989(16)
C(17A)-H(17B)	1.004(13)	C(9B)-H(9E)	1.005(18)
C(17A)-H(17C)	0.973(14)	C(9B)-H(9F)	0.974(18)
C(18A)-H(18A)	0.988(18)	C(10B)-H(10D)	0.976(14)
C(18A)-H(18B)	0.957(16)	C(10B)-H(10E)	0.961(14)
C(18A)-H(18C)	0.98(2)	C(10B)-H(10F)	1.025(15)
C(19A)-H(19A)	1.003(13)	C(11B)-C(16B)	1.4002(18)
C(19A)-H(19B)	0.976(14)	C(11B)-C(12B)	1.4035(18)
C(19A)-H(19C)	0.986(15)	C(12B)-C(13B)	1.3922(18)

C(12B)-C(17B)	1.502(2)	C(1B)-C(1A)-P(1A)	168.05(10)
C(13B)-C(14B)	1.391(2)	N(1A)-C(1A)-P(1A)	48.06(6)
C(13B)-H(13B)	0.989(13)	N(2A)-C(1A)-P(1A)	48.27(6)
C(14B)-C(15B)	1.376(2)	C(3A)-C(2A)-C(7A)	119.37(12)
C(14B)-C(18B)	1.515(2)	C(3A)-C(2A)-N(1A)	119.15(12)
C(15B)-C(16B)	1.3929(19)	C(7A)-C(2A)-N(1A)	121.40(12)
C(15B)-H(15B)	0.941(14)	C(4A)-C(3A)-C(2A)	119.17(13)
C(16B)-C(19B)	1.497(2)	C(4A)-C(3A)-C(8A)	118.28(13)
C(17B)-H(17D)	0.974(14)	C(2A)-C(3A)-C(8A)	122.54(12)
C(17B)-H(17E)	0.991(13)	C(5A)-C(4A)-C(3A)	122.44(14)
C(17B)-H(17F)	1.003(15)	C(5A)-C(4A)-H(4A)	121.4(7)
C(18B)-H(18D)	0.97(2)	C(3A)-C(4A)-H(4A)	116.2(7)
C(18B)-H(18E)	0.992(18)	C(6A)-C(5A)-C(4A)	117.12(13)
C(18B)-H(18F)	0.964(19)	C(6A)-C(5A)-C(9A)	120.94(14)
C(19B)-H(19D)	0.959(17)	C(4A)-C(5A)-C(9A)	121.94(14)
C(19B)-H(19E)	0.986(15)	C(5A)-C(6A)-C(7A)	123.12(14)
C(19B)-H(19F)	0.944(17)	C(5A)-C(6A)-H(6A)	119.8(7)
C(20B)-C(21B)	1.518(2)	C(7A)-C(6A)-H(6A)	117.0(7)
C(20B)-C(22B)	1.522(2)	C(6A)-C(7A)-C(2A)	118.69(13)
C(20B)-H(20B)	0.997(14)	C(6A)-C(7A)-C(10A)	118.42(13)
C(21B)-H(21D)	0.987(17)	C(2A)-C(7A)-C(10A)	122.82(12)
C(21B)-H(21E)	1.001(16)	C(3A)-C(8A)-H(8A)	114.3(9)
C(21B)-H(21F)	0.950(19)	C(3A)-C(8A)-H(8B)	113.2(9)
C(22B)-H(22D)	1.004(18)	H(8A)-C(8A)-H(8B)	102.7(12)
C(22B)-H(22E)	0.998(15)	C(3A)-C(8A)-H(8C)	111.9(8)
C(22B)-H(22F)	0.948(19)	H(8A)-C(8A)-H(8C)	105.7(12)
C(23B)-C(25B)	1.516(2)	H(8B)-C(8A)-H(8C)	108.3(12)
C(23B)-C(24B)	1.523(2)	C(5A)-C(9A)-H(9A)	113.2(11)
C(23B)-H(23B)	0.971(13)	C(5A)-C(9A)-H(9B)	113.3(10)
C(24B)-H(24D)	1.013(18)	H(9A)-C(9A)-H(9B)	111.1(15)
C(24B)-H(24E)	0.936(19)	C(5A)-C(9A)-H(9C)	111.5(10)
C(24B)-H(24F)	1.00(2)	H(9A)-C(9A)-H(9C)	103.8(15)
C(25B)-H(25D)	0.976(15)	H(9B)-C(9A)-H(9C)	103.0(14)
C(25B)-H(25E)	0.976(17)	C(7A)-C(10A)-H(10A)	110.9(8)
C(25B)-H(25F)	1.013(16)	C(7A)-C(10A)-H(10B)	114.7(8)
		H(10A)-C(10A)-H(10B)	106.7(11)
N(3A)-P(1A)-N(1A)	106.68(5)	C(7A)-C(10A)-H(10C)	111.4(9)
N(3A)-P(1A)-N(2A)	110.44(5)	H(10A)-C(10A)-H(10C)	109.1(12)
N(1A)-P(1A)-N(2A)	75.02(5)	H(10B)-C(10A)-H(10C)	103.6(11)
N(3A)-P(1A)-C(1A)	119.25(5)	C(16A)-C(11A)-C(12A)	120.10(12)
N(1A)-P(1A)-C(1A)	37.63(4)	C(16A)-C(11A)-N(2A)	123.16(12)
N(2A)-P(1A)-C(1A)	38.11(4)	C(12A)-C(11A)-N(2A)	116.55(12)
C(2A)-N(1A)-C(1A)	134.14(10)	C(13A)-C(12A)-C(11A)	118.73(13)
C(2A)-N(1A)-P(1A)	131.37(9)	C(13A)-C(12A)-C(17A)	119.74(13)
C(1A)-N(1A)-P(1A)	94.32(8)	C(11A)-C(12A)-C(17A)	121.52(12)
C(11A)-N(2A)-C(1A)	127.79(10)	C(14A)-C(13A)-C(12A)	122.25(13)
C(11A)-N(2A)-P(1A)	120.18(8)	C(14A)-C(13A)-H(13A)	120.1(7)
C(1A)-N(2A)-P(1A)	93.62(8)	C(12A)-C(13A)-H(13A)	117.7(7)
C(20A)-N(3A)-C(23A)	116.31(11)	C(15A)-C(14A)-C(13A)	117.81(13)
C(20A)-N(3A)-P(1A)	125.69(9)	C(15A)-C(14A)-C(18A)	121.10(15)
C(23A)-N(3A)-P(1A)	117.82(9)	C(13A)-C(14A)-C(18A)	121.09(14)
C(1B)-C(1A)-N(1A)	134.74(12)	C(14A)-C(15A)-C(16A)	122.46(14)
C(1B)-C(1A)-N(2A)	129.75(12)	C(14A)-C(15A)-H(15A)	117.6(8)
N(1A)-C(1A)-N(2A)	95.30(10)	C(16A)-C(15A)-H(15A)	119.9(8)

C(15A)-C(16A)-C(11A)	118.48(13)	C(23A)-C(25A)-H(25C)	107.9(8)
C(15A)-C(16A)-C(19A)	119.35(13)	H(25A)-C(25A)-H(25C)	110.1(12)
C(11A)-C(16A)-C(19A)	122.18(13)	H(25B)-C(25A)-H(25C)	109.3(11)
C(12A)-C(17A)-H(17A)	110.8(8)	N(3B)-P(1B)-N(1B)	107.08(6)
C(12A)-C(17A)-H(17B)	111.5(7)	N(3B)-P(1B)-N(2B)	110.51(6)
H(17A)-C(17A)-H(17B)	107.7(11)	N(1B)-P(1B)-N(2B)	74.82(5)
C(12A)-C(17A)-H(17C)	110.5(8)	N(3B)-P(1B)-C(1B)	119.63(5)
H(17A)-C(17A)-H(17C)	105.2(11)	N(1B)-P(1B)-C(1B)	37.55(4)
H(17B)-C(17A)-H(17C)	110.9(11)	N(2B)-P(1B)-C(1B)	38.02(4)
C(14A)-C(18A)-H(18A)	113.8(9)	C(2B)-N(1B)-C(1B)	132.99(11)
C(14A)-C(18A)-H(18B)	110.0(10)	C(2B)-N(1B)-P(1B)	132.22(9)
H(18A)-C(18A)-H(18B)	105.6(13)	C(1B)-N(1B)-P(1B)	94.66(8)
C(14A)-C(18A)-H(18C)	112.5(11)	C(11B)-N(2B)-C(1B)	125.49(10)
H(18A)-C(18A)-H(18C)	107.2(15)	C(11B)-N(2B)-P(1B)	121.68(8)
H(18B)-C(18A)-H(18C)	107.4(14)	C(1B)-N(2B)-P(1B)	93.58(8)
C(16A)-C(19A)-H(19A)	113.3(7)	C(20B)-N(3B)-C(23B)	116.52(12)
C(16A)-C(19A)-H(19B)	112.7(8)	C(20B)-N(3B)-P(1B)	125.39(10)
H(19A)-C(19A)-H(19B)	106.2(11)	C(23B)-N(3B)-P(1B)	117.91(10)
C(16A)-C(19A)-H(19C)	111.2(8)	C(1A)-C(1B)-N(1B)	134.63(12)
H(19A)-C(19A)-H(19C)	107.1(11)	C(1A)-C(1B)-N(2B)	130.02(12)
H(19B)-C(19A)-H(19C)	105.9(11)	N(1B)-C(1B)-N(2B)	95.12(10)
N(3A)-C(20A)-C(22A)	112.94(12)	C(1A)-C(1B)-P(1B)	167.84(10)
N(3A)-C(20A)-C(21A)	111.24(12)	N(1B)-C(1B)-P(1B)	47.79(6)
C(22A)-C(20A)-C(21A)	110.64(13)	N(2B)-C(1B)-P(1B)	48.40(6)
N(3A)-C(20A)-H(20A)	104.9(7)	C(7B)-C(2B)-C(3B)	119.76(12)
C(22A)-C(20A)-H(20A)	107.0(7)	C(7B)-C(2B)-N(1B)	120.92(12)
C(21A)-C(20A)-H(20A)	109.9(7)	C(3B)-C(2B)-N(1B)	119.27(12)
C(20A)-C(21A)-H(21A)	114.0(8)	C(4B)-C(3B)-C(2B)	118.61(13)
C(20A)-C(21A)-H(21B)	112.1(8)	C(4B)-C(3B)-C(8B)	118.50(13)
H(21A)-C(21A)-H(21B)	107.3(11)	C(2B)-C(3B)-C(8B)	122.86(13)
C(20A)-C(21A)-H(21C)	110.2(8)	C(5B)-C(4B)-C(3B)	122.56(14)
H(21A)-C(21A)-H(21C)	107.5(11)	C(5B)-C(4B)-H(4B)	120.6(8)
H(21B)-C(21A)-H(21C)	105.3(11)	C(3B)-C(4B)-H(4B)	116.8(8)
C(20A)-C(22A)-H(22A)	113.0(8)	C(4B)-C(5B)-C(6B)	117.49(13)
C(20A)-C(22A)-H(22B)	112.0(8)	C(4B)-C(5B)-C(9B)	121.89(14)
H(22A)-C(22A)-H(22B)	108.3(11)	C(6B)-C(5B)-C(9B)	120.62(15)
C(20A)-C(22A)-H(22C)	111.1(8)	C(7B)-C(6B)-C(5B)	122.73(14)
H(22A)-C(22A)-H(22C)	105.3(12)	C(7B)-C(6B)-H(6B)	118.8(7)
H(22B)-C(22A)-H(22C)	106.8(12)	C(5B)-C(6B)-H(6B)	118.5(7)
N(3A)-C(23A)-C(24A)	113.22(12)	C(6B)-C(7B)-C(2B)	118.77(13)
N(3A)-C(23A)-C(25A)	111.74(12)	C(6B)-C(7B)-C(10B)	119.05(13)
C(24A)-C(23A)-C(25A)	112.07(14)	C(2B)-C(7B)-C(10B)	122.17(12)
N(3A)-C(23A)-H(23A)	103.8(7)	C(3B)-C(8B)-H(8D)	109.5(11)
C(24A)-C(23A)-H(23A)	107.2(7)	C(3B)-C(8B)-H(8E)	114.9(9)
C(25A)-C(23A)-H(23A)	108.2(7)	H(8D)-C(8B)-H(8E)	106.7(14)
C(23A)-C(24A)-H(24A)	110.2(8)	C(3B)-C(8B)-H(8F)	113.4(10)
C(23A)-C(24A)-H(24B)	109.0(8)	H(8D)-C(8B)-H(8F)	109.6(14)
H(24A)-C(24A)-H(24B)	109.5(11)	H(8E)-C(8B)-H(8F)	102.4(13)
C(23A)-C(24A)-H(24C)	111.5(8)	C(5B)-C(9B)-H(9D)	110.3(9)
H(24A)-C(24A)-H(24C)	109.5(12)	C(5B)-C(9B)-H(9E)	111.6(10)
H(24B)-C(24A)-H(24C)	107.1(11)	H(9D)-C(9B)-H(9E)	103.8(13)
C(23A)-C(25A)-H(25A)	111.2(9)	C(5B)-C(9B)-H(9F)	113.3(10)
C(23A)-C(25A)-H(25B)	112.0(8)	H(9D)-C(9B)-H(9F)	112.4(14)
H(25A)-C(25A)-H(25B)	106.3(12)	H(9E)-C(9B)-H(9F)	104.8(14)

C(7B)-C(10B)-H(10D)	111.5(8)	C(16B)-C(19B)-H(19F)	113.8(10)
C(7B)-C(10B)-H(10E)	113.0(8)	H(19D)-C(19B)-H(19F)	109.9(14)
H(10D)-C(10B)-H(10E)	106.9(11)	H(19E)-C(19B)-H(19F)	103.4(13)
C(7B)-C(10B)-H(10F)	111.8(7)	N(3B)-C(20B)-C(21B)	111.44(15)
H(10D)-C(10B)-H(10F)	107.7(11)	N(3B)-C(20B)-C(22B)	113.48(14)
H(10E)-C(10B)-H(10F)	105.5(11)	C(21B)-C(20B)-C(22B)	110.67(15)
C(16B)-C(11B)-C(12B)	119.97(12)	N(3B)-C(20B)-H(20B)	103.9(8)
C(16B)-C(11B)-N(2B)	117.28(12)	C(21B)-C(20B)-H(20B)	110.7(8)
C(12B)-C(11B)-N(2B)	122.68(12)	C(22B)-C(20B)-H(20B)	106.3(8)
C(13B)-C(12B)-C(11B)	118.72(13)	C(20B)-C(21B)-H(21D)	114.0(9)
C(13B)-C(12B)-C(17B)	119.25(14)	C(20B)-C(21B)-H(21E)	113.2(9)
C(11B)-C(12B)-C(17B)	122.02(12)	H(21D)-C(21B)-H(21E)	100.7(13)
C(14B)-C(13B)-C(12B)	122.04(15)	C(20B)-C(21B)-H(21F)	112.2(11)
C(14B)-C(13B)-H(13B)	120.4(8)	H(21D)-C(21B)-H(21F)	108.6(14)
C(12B)-C(13B)-H(13B)	117.6(8)	H(21E)-C(21B)-H(21F)	107.4(14)
C(15B)-C(14B)-C(13B)	117.79(14)	C(20B)-C(22B)-H(22D)	111.9(10)
C(15B)-C(14B)-C(18B)	121.80(16)	C(20B)-C(22B)-H(22E)	113.1(9)
C(13B)-C(14B)-C(18B)	120.41(16)	H(22D)-C(22B)-H(22E)	108.7(13)
C(14B)-C(15B)-C(16B)	122.49(15)	C(20B)-C(22B)-H(22F)	110.1(11)
C(14B)-C(15B)-H(15B)	120.1(8)	H(22D)-C(22B)-H(22F)	108.2(14)
C(16B)-C(15B)-H(15B)	117.4(8)	H(22E)-C(22B)-H(22F)	104.6(13)
C(15B)-C(16B)-C(11B)	118.68(14)	N(3B)-C(23B)-C(25B)	111.59(13)
C(15B)-C(16B)-C(19B)	120.01(14)	N(3B)-C(23B)-C(24B)	112.35(14)
C(11B)-C(16B)-C(19B)	121.31(13)	C(25B)-C(23B)-C(24B)	111.60(17)
C(12B)-C(17B)-H(17D)	112.4(8)	N(3B)-C(23B)-H(23B)	106.3(8)
C(12B)-C(17B)-H(17E)	110.7(8)	C(25B)-C(23B)-H(23B)	109.7(8)
H(17D)-C(17B)-H(17E)	108.9(11)	C(24B)-C(23B)-H(23B)	105.0(7)
C(12B)-C(17B)-H(17F)	109.5(8)	C(23B)-C(24B)-H(24D)	108.4(10)
H(17D)-C(17B)-H(17F)	105.1(11)	C(23B)-C(24B)-H(24E)	110.0(11)
H(17E)-C(17B)-H(17F)	110.0(11)	H(24D)-C(24B)-H(24E)	108.7(14)
C(14B)-C(18B)-H(18D)	110.2(11)	C(23B)-C(24B)-H(24F)	110.9(12)
C(14B)-C(18B)-H(18E)	110.9(10)	H(24D)-C(24B)-H(24F)	110.1(16)
H(18D)-C(18B)-H(18E)	107.7(15)	H(24E)-C(24B)-H(24F)	108.8(16)
C(14B)-C(18B)-H(18F)	111.5(10)	C(23B)-C(25B)-H(25D)	111.7(9)
H(18D)-C(18B)-H(18F)	109.0(16)	C(23B)-C(25B)-H(25E)	110.6(9)
H(18E)-C(18B)-H(18F)	107.5(14)	H(25D)-C(25B)-H(25E)	107.3(12)
C(16B)-C(19B)-H(19D)	111.6(10)	C(23B)-C(25B)-H(25F)	110.4(9)
C(16B)-C(19B)-H(19E)	111.0(9)	H(25D)-C(25B)-H(25F)	109.2(12)
H(19D)-C(19B)-H(19E)	106.7(12)	H(25E)-C(25B)-H(25F)	107.6(13)

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for 4. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

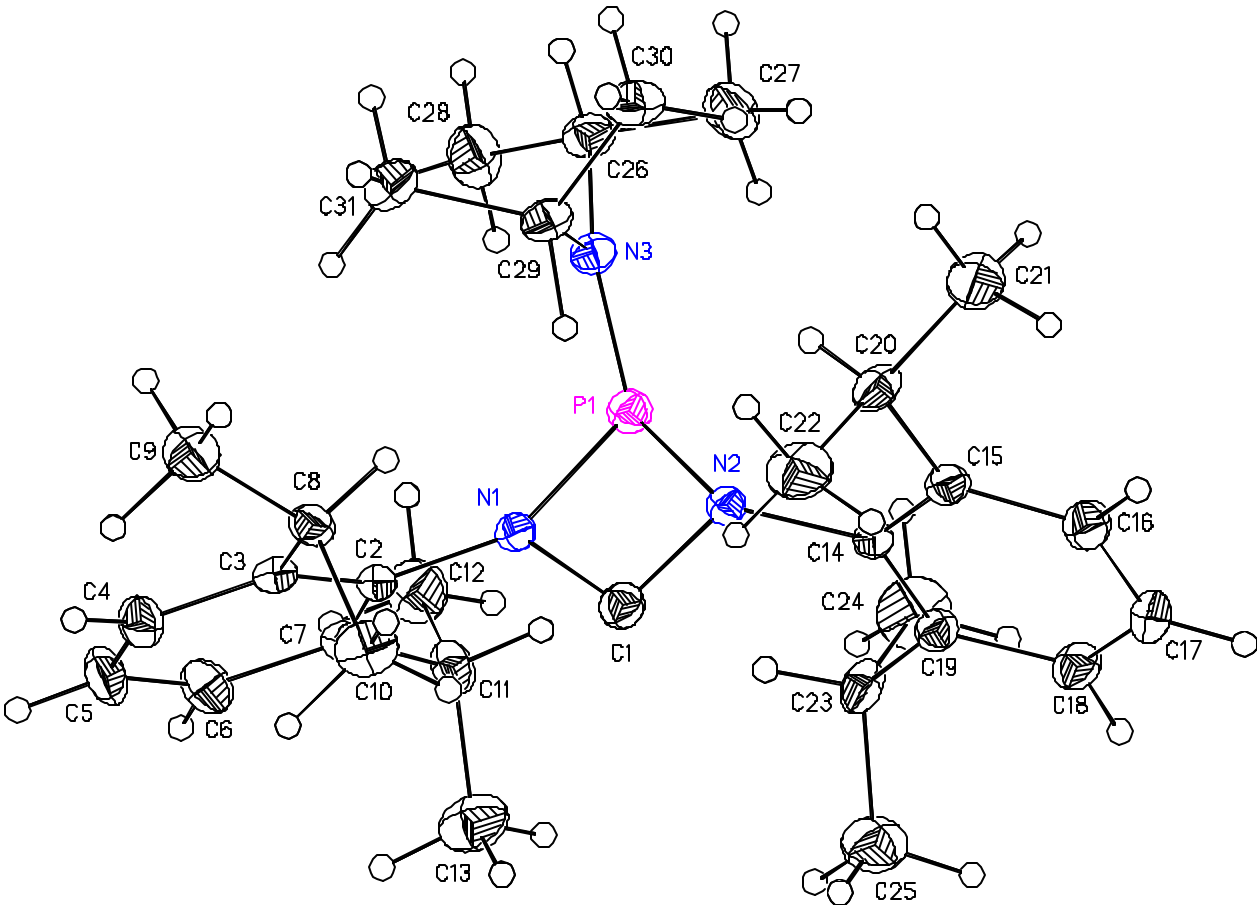
	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P(1A)	156(2)	154(2)	148(2)	-12(2)	38(2)	-17(2)
N(1A)	142(6)	170(6)	127(6)	-37(5)	30(5)	-32(5)
N(2A)	122(6)	151(6)	157(6)	-28(5)	36(5)	-26(5)
N(3A)	203(6)	155(6)	141(6)	-22(5)	67(5)	-5(5)
C(1A)	127(7)	130(7)	163(7)	6(6)	17(6)	-7(6)
C(2A)	127(7)	135(7)	189(8)	-41(6)	31(6)	-3(6)
C(3A)	168(7)	160(7)	193(8)	-22(6)	42(6)	11(6)
C(4A)	205(8)	231(9)	196(8)	-71(7)	-15(7)	11(7)
C(5A)	166(8)	215(8)	299(9)	-50(7)	29(7)	-41(6)
C(6A)	203(8)	180(8)	274(9)	2(7)	86(7)	-31(6)
C(7A)	165(7)	149(7)	191(8)	-24(6)	47(6)	5(6)
C(8A)	203(8)	263(9)	173(8)	8(7)	5(7)	21(7)
C(9A)	225(9)	361(11)	440(12)	-52(10)	6(8)	-114(9)
C(10A)	224(9)	206(9)	206(9)	20(7)	52(7)	-39(7)
C(11A)	171(7)	184(8)	131(7)	-39(6)	53(6)	-36(6)
C(12A)	164(7)	211(8)	163(7)	-32(6)	55(6)	-9(6)
C(13A)	139(8)	290(9)	210(8)	-42(7)	34(6)	-37(7)
C(14A)	225(8)	245(9)	207(8)	-16(7)	53(7)	-101(7)
C(15A)	286(9)	172(8)	219(8)	13(7)	81(7)	-43(7)
C(16A)	195(8)	200(8)	164(7)	-10(6)	54(6)	-22(6)
C(17A)	163(8)	223(9)	240(9)	-41(7)	31(7)	2(7)
C(18A)	320(11)	327(11)	409(12)	22(10)	33(9)	-165(9)
C(19A)	238(9)	181(9)	230(9)	37(7)	40(7)	-17(7)
C(20A)	274(8)	163(8)	193(8)	-8(7)	96(7)	12(6)
C(21A)	272(9)	186(9)	342(10)	5(8)	98(8)	-24(7)
C(22A)	328(10)	185(9)	217(9)	13(7)	32(8)	24(7)
C(23A)	216(8)	182(8)	177(8)	-44(7)	73(6)	1(6)
C(24A)	286(9)	314(10)	185(9)	-46(8)	65(7)	10(8)
C(25A)	233(9)	406(12)	307(10)	-116(9)	137(8)	-49(8)
P(1B)	170(2)	195(2)	162(2)	-21(2)	41(2)	17(2)
N(1B)	133(6)	193(7)	140(6)	-28(5)	34(5)	0(5)
N(2B)	135(6)	162(6)	142(6)	-21(5)	18(5)	5(5)
N(3B)	254(7)	233(7)	193(7)	-32(6)	47(5)	86(6)
C(1B)	122(7)	143(7)	153(7)	9(6)	12(6)	-10(6)
C(2B)	144(7)	168(8)	202(8)	-51(6)	48(6)	11(6)
C(3B)	190(8)	192(8)	217(8)	-39(6)	82(6)	-1(6)
C(4B)	203(8)	246(9)	298(9)	-59(7)	127(7)	-55(7)
C(5B)	148(7)	265(9)	302(9)	-91(7)	43(7)	-14(6)
C(6B)	186(8)	261(9)	184(8)	-38(7)	9(7)	44(6)
C(7B)	161(7)	167(8)	196(8)	-37(6)	49(6)	37(6)
C(8B)	286(10)	271(10)	262(9)	20(8)	113(8)	-31(8)
C(9B)	161(9)	471(13)	393(12)	-112(10)	37(8)	-69(8)
C(10B)	184(8)	227(9)	174(8)	19(7)	6(7)	27(7)
C(11B)	147(7)	220(8)	114(7)	-49(6)	35(6)	-5(6)
C(12B)	198(8)	250(9)	119(7)	-41(6)	36(6)	32(6)
C(13B)	230(8)	317(10)	165(8)	-34(7)	16(6)	84(7)
C(14B)	176(8)	414(11)	179(8)	-106(7)	1(6)	28(7)
C(15B)	216(8)	303(10)	238(9)	-119(7)	52(7)	-65(7)

C(16B)	184(8)	236(8)	177(8)	-63(6)	38(6)	-19(6)
C(17B)	233(9)	205(9)	211(9)	16(7)	29(7)	58(7)
C(18B)	208(9)	598(15)	392(12)	-169(11)	-40(9)	36(9)
C(19B)	270(9)	226(9)	344(11)	-61(8)	41(9)	-66(8)
C(20B)	422(11)	279(10)	240(9)	-14(8)	46(8)	169(8)
C(21B)	476(13)	375(12)	360(12)	-91(10)	-114(10)	211(10)
C(22B)	530(14)	287(11)	403(12)	45(9)	117(11)	145(9)
C(23B)	296(9)	255(9)	274(9)	-78(7)	96(7)	69(7)
C(24B)	624(16)	489(15)	685(16)	-251(13)	505(14)	-93(12)
C(25B)	477(12)	313(11)	251(10)	-78(9)	20(9)	92(10)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 4.

	x	y	z	U_{iso}
H(4A)	-9(9)	1825(8)	-871(6)	14(3)
H(6A)	233(9)	960(8)	924(6)	16(4)
H(8A)	2105(11)	2547(9)	-898(7)	37(5)
H(8B)	1750(11)	3316(10)	-607(7)	41(5)
H(8C)	1019(10)	2817(8)	-1128(7)	28(4)
H(9A)	-1611(15)	1199(12)	-203(9)	73(7)
H(9B)	-1057(12)	360(12)	64(8)	61(6)
H(9C)	-1107(12)	629(11)	-622(9)	65(6)
H(10A)	1503(10)	1279(8)	1719(6)	27(4)
H(10B)	2150(9)	2098(9)	1697(6)	26(4)
H(10C)	2517(11)	1281(9)	1467(7)	37(5)
H(13A)	7163(9)	4024(8)	1576(6)	18(4)
H(15A)	5189(9)	5740(9)	770(6)	22(4)
H(17A)	6103(9)	2377(9)	1108(7)	26(4)
H(17B)	5499(10)	2439(8)	1675(6)	20(4)
H(17C)	6663(11)	2692(9)	1766(7)	32(4)
H(18A)	6900(11)	6168(11)	1092(8)	52(5)
H(18B)	7666(12)	5458(10)	1232(7)	43(5)
H(18C)	7221(13)	5819(11)	1783(10)	73(7)
H(19A)	3059(9)	4956(8)	765(6)	24(4)
H(19B)	3229(9)	4464(8)	165(6)	21(4)
H(19C)	3559(10)	5388(9)	242(7)	30(4)
H(20A)	3814(9)	1413(8)	1036(6)	16(4)
H(21A)	2617(10)	650(8)	265(6)	25(4)
H(21B)	3496(9)	92(9)	85(7)	27(4)
H(21C)	3221(10)	36(9)	771(7)	36(4)
H(22A)	5350(10)	426(9)	645(7)	29(4)
H(22B)	5521(10)	1137(9)	1190(7)	35(4)
H(22C)	4968(10)	302(9)	1273(7)	31(4)
H(23A)	4651(8)	861(8)	-263(6)	11(3)
H(24A)	3149(11)	1262(9)	-927(7)	34(4)
H(24B)	4067(9)	1227(8)	-1296(6)	23(4)
H(24C)	3681(10)	2082(10)	-1094(6)	29(4)
H(25A)	5405(11)	2482(11)	-430(7)	45(5)
H(25B)	5785(10)	1677(9)	-715(7)	28(4)
H(25C)	5974(10)	1758(9)	45(7)	31(4)
H(4B)	-1249(9)	2515(8)	1567(6)	19(4)
H(6B)	-793(8)	3692(7)	4(6)	9(3)
H(8D)	340(12)	3103(11)	2606(9)	62(6)
H(8E)	1139(12)	2551(10)	2430(7)	47(5)
H(8F)	128(12)	2199(11)	2397(8)	51(5)
H(9D)	-2708(12)	3262(10)	473(7)	48(5)
H(9E)	-2454(12)	2319(12)	541(8)	58(6)
H(9F)	-2266(12)	2766(11)	-65(9)	60(6)
H(10D)	663(10)	4367(8)	-56(7)	27(4)
H(10E)	1633(10)	4008(8)	345(6)	24(4)
H(10F)	1184(9)	4813(9)	591(6)	28(4)

H(13B)	5816(9)	2671(9)	3030(6)	22(4)
H(15B)	6159(10)	5129(9)	2781(6)	25(4)
H(17D)	3895(9)	2209(9)	2134(7)	27(4)
H(17E)	3253(10)	2645(8)	2581(6)	22(4)
H(17F)	4238(10)	2100(9)	2863(7)	32(4)
H(18D)	7362(13)	3471(12)	3593(10)	73(7)
H(18E)	7577(12)	4356(11)	3337(8)	59(6)
H(18F)	7629(13)	3566(11)	2922(9)	65(6)
H(19D)	4010(11)	5789(10)	2529(8)	47(5)
H(19E)	3901(11)	5539(9)	1824(7)	33(4)
H(19F)	4850(12)	5948(11)	2156(8)	53(5)
H(20B)	1793(10)	5326(9)	1497(7)	29(4)
H(21D)	15(11)	5066(11)	1475(7)	40(5)
H(21E)	-60(11)	5864(10)	1839(8)	48(5)
H(21F)	172(13)	5915(11)	1159(9)	65(6)
H(22D)	2584(14)	6455(10)	2084(8)	59(6)
H(22E)	1520(10)	6790(9)	2200(7)	35(4)
H(22F)	1734(12)	6758(11)	1530(9)	60(6)
H(23B)	928(9)	5832(8)	2816(6)	17(4)
H(24D)	107(13)	4603(11)	2845(9)	58(6)
H(24E)	451(12)	4969(11)	3530(9)	62(6)
H(24F)	1066(15)	4225(14)	3342(10)	86(8)
H(25D)	2658(10)	5144(10)	3592(7)	32(4)
H(25E)	2003(11)	5838(10)	3810(8)	48(5)
H(25F)	2627(11)	6054(10)	3279(7)	45(5)



5

Special Refinement Details

Refinement of F^2 against ALL reflections. The weighted R-factor (*w*R) and goodness of fit (S) are based on F^2, conventional R-factors (R) are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\sigma(F^2)$ is used only for calculating R-factors(gt) etc. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R-factors based on ALL data will be even larger.

All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Table 1. Crystal data and structure refinement for 5.

Identification code	**5**
Empirical formula	$C_{31}H_{48}N_3P$
Formula weight	493.69
Crystal size	0.41 x 0.22 x 0.07 mm^3
Crystal color	Colorless
Wavelength	0.71073 Å MoKα
Data Collection Temperature	100(2) K
Unit cell dimensions	a = 8.9994(10) Å b = 26.366(3) Å β= 94.240(2)° c = 12.6593(14) Å
Volume	2995.5(6) $Å^3$
Z	4
Crystal system	Monoclinic
Space group	$P2_1/n$
Density (calculated)	1.095 Mg/m^3
F(000)	1080
θ range for data collection	1.79 to 28.38°
Completeness to θ = 28.38°	92.1 %
Index ranges	$-11 \le h \le 11$, $-34 \le k \le 33$, $-16 \le l \le 15$
Reflections collected	26662
Independent reflections	6905 [R_{int}= 0.0643]
Absorption coefficient	0.114 mm^{-1}
Absorption correction	None
Max. and min. transmission	0.9921 and 0.9547
Refinement method	Full matrix least-squares on F^2
Data / restraints / parameters	6905 / 0 / 508
Goodness-of-fit on F^2	1.496
Final R indices [$I>2\sigma(I)$, 4684 reflections]	R1 = 0.0525, *w*R2 = 0.0810
R indices (all data)	R1 = 0.0822, *w*R2 = 0.0846
Largest diff. peak and hole	1.025 and -0.551 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 5. U(eq) is defined as the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U_{eq}
P(1)	8866(1)	1379(1)	7257(1)	19(1)
N(1)	7306(1)	1418(1)	8029(1)	16(1)
N(2)	7625(1)	876(1)	6894(1)	16(1)
N(3)	10296(1)	1167(1)	8011(1)	18(1)
C(1)	6464(2)	1078(1)	7419(1)	20(1)
C(2)	6751(2)	1796(1)	8713(1)	17(1)
C(3)	6447(2)	1653(1)	9748(1)	18(1)
C(4)	5939(2)	2024(1)	10406(2)	25(1)
C(5)	5713(2)	2514(1)	10073(2)	29(1)
C(6)	5979(2)	2648(1)	9049(2)	26(1)
C(7)	6484(2)	2294(1)	8344(1)	20(1)
C(8)	6578(2)	1107(1)	10123(1)	20(1)
C(9)	6996(3)	1052(1)	11300(2)	30(1)
C(10)	5116(2)	824(1)	9829(2)	30(1)
C(11)	6637(2)	2455(1)	7203(1)	23(1)
C(12)	7758(3)	2882(1)	7118(2)	35(1)
C(13)	5132(2)	2607(1)	6658(2)	37(1)
C(14)	7365(2)	587(1)	5929(1)	17(1)
C(15)	7671(2)	66(1)	5965(1)	19(1)
C(16)	7319(2)	-218(1)	5055(2)	25(1)
C(17)	6691(2)	5(1)	4142(2)	25(1)
C(18)	6443(2)	518(1)	4118(2)	23(1)
C(19)	6759(2)	822(1)	5005(1)	18(1)
C(20)	8310(2)	-184(1)	6977(2)	23(1)
C(21)	9298(3)	-638(1)	6790(2)	34(1)
C(22)	7083(3)	-322(1)	7690(2)	35(1)
C(23)	6476(2)	1387(1)	4920(2)	24(1)
C(24)	7643(3)	1638(1)	4282(2)	42(1)
C(25)	4912(3)	1516(1)	4466(2)	37(1)
C(26)	11787(2)	1412(1)	7921(2)	23(1)
C(27)	12342(2)	1328(1)	6829(2)	30(1)
C(28)	11790(3)	1969(1)	8228(2)	33(1)
C(29)	10196(2)	794(1)	8878(1)	22(1)
C(30)	11281(2)	352(1)	8786(2)	26(1)
C(31)	10423(2)	1051(1)	9952(2)	26(1)

Table 3. Bond lengths [Å] and angles [°] for 5.

P(1)-N(3)	1.6429(14)	C(21)-H(21A)	0.99(2)
P(1)-N(1)	1.7720(13)	C(21)-H(21B)	0.970(19)
P(1)-N(2)	1.7731(14)	C(21)-H(21C)	0.948(19)
N(1)-C(1)	1.373(2)	C(22)-H(22A)	0.959(19)
N(1)-C(2)	1.435(2)	C(22)-H(22B)	0.99(2)
N(2)-C(1)	1.387(2)	C(22)-H(22C)	1.029(19)
N(2)-C(14)	1.443(2)	C(23)-C(25)	1.519(3)
N(3)-C(29)	1.481(2)	C(23)-C(24)	1.523(3)
N(3)-C(26)	1.501(2)	C(23)-H(23)	0.921(16)
C(2)-C(7)	1.406(2)	C(24)-H(24A)	1.01(2)
C(2)-C(3)	1.410(2)	C(24)-H(24B)	0.98(2)
C(3)-C(4)	1.386(2)	C(24)-H(24C)	0.982(19)
C(3)-C(8)	1.518(2)	C(25)-H(25A)	1.02(2)
C(4)-C(5)	1.370(3)	C(25)-H(25B)	0.95(2)
C(4)-H(4)	0.926(17)	C(25)-H(25C)	0.97(2)
C(5)-C(6)	1.381(3)	C(26)-C(28)	1.520(3)
C(5)-H(5)	0.920(17)	C(26)-C(27)	1.519(3)
C(6)-C(7)	1.391(2)	C(26)-H(26)	1.046(16)
C(6)-H(6)	0.970(16)	C(27)-H(27A)	0.98(2)
C(7)-C(11)	1.522(2)	C(27)-H(27B)	0.981(19)
C(8)-C(9)	1.517(3)	C(27)-H(27C)	0.991(19)
C(8)-C(10)	1.534(3)	C(28)-H(28A)	0.985(17)
C(8)-H(8)	0.952(15)	C(28)-H(28B)	0.98(2)
C(9)-H(9A)	0.940(19)	C(28)-H(28C)	1.018(19)
C(9)-H(9B)	0.976(19)	C(29)-C(31)	1.519(2)
C(9)-H(9C)	1.024(18)	C(29)-C(30)	1.531(2)
C(10)-H(10A)	1.011(19)	C(29)-H(29)	1.098(16)
C(10)-H(10B)	0.955(19)	C(30)-H(30A)	0.969(17)
C(10)-H(10C)	0.960(17)	C(30)-H(30B)	0.997(19)
C(11)-C(12)	1.520(3)	C(30)-H(30C)	0.901(17)
C(11)-C(13)	1.527(3)	C(31)-H(31A)	0.964(16)
C(11)-H(11)	0.966(15)	C(31)-H(31B)	0.982(19)
C(12)-H(12A)	0.992(19)	C(31)-H(31C)	0.99(2)
C(12)-H(12B)	0.986(19)	N(3)-P(1)-N(1)	108.53(7)
C(12)-H(12C)	0.93(2)	N(3)-P(1)-N(2)	110.30(7)
C(13)-H(13A)	1.04(2)	N(1)-P(1)-N(2)	71.13(6)
C(13)-H(13B)	1.024(19)	N(3)-P(1)-C(1)	121.72(7)
C(13)-H(13C)	0.917(19)	N(1)-P(1)-C(1)	36.04(6)
C(14)-C(19)	1.398(2)	N(2)-P(1)-C(1)	36.49(6)
C(14)-C(15)	1.402(2)	C(1)-N(1)-C(2)	126.21(13)
C(15)-C(16)	1.391(2)	C(1)-N(1)-P(1)	94.54(10)
C(15)-C(20)	1.515(2)	C(2)-N(1)-P(1)	134.31(11)
C(16)-C(17)	1.380(3)	C(1)-N(2)-C(14)	121.87(13)
C(16)-H(16)	0.968(18)	C(1)-N(2)-P(1)	93.99(10)
C(17)-C(18)	1.372(3)	C(14)-N(2)-P(1)	132.34(11)
C(17)-H(17)	0.939(18)	C(29)-N(3)-C(26)	116.46(13)
C(18)-C(19)	1.393(2)	C(29)-N(3)-P(1)	124.68(11)
C(18)-H(18)	0.963(16)	C(26)-N(3)-P(1)	118.39(11)
C(19)-C(23)	1.513(2)	N(1)-C(1)-N(2)	96.72(13)
C(20)-C(21)	1.521(3)	N(1)-C(1)-P(1)	49.42(8)
C(20)-C(22)	1.521(3)	N(2)-C(1)-P(1)	49.51(8)
C(20)-H(20)	0.917(15)	C(7)-C(2)-C(3)	121.27(15)

C(7)-C(2)-N(1)	120.39(15)	C(19)-C(14)-N(2)	120.22(15)
C(3)-C(2)-N(1)	118.32(15)	C(15)-C(14)-N(2)	118.07(15)
C(4)-C(3)-C(2)	117.66(16)	C(16)-C(15)-C(14)	118.03(16)
C(4)-C(3)-C(8)	120.26(16)	C(16)-C(15)-C(20)	120.97(16)
C(2)-C(3)-C(8)	121.99(15)	C(14)-C(15)-C(20)	120.95(15)
C(5)-C(4)-C(3)	121.96(18)	C(17)-C(16)-C(15)	121.21(18)
C(5)-C(4)-H(4)	119.4(11)	C(17)-C(16)-H(16)	121.5(11)
C(3)-C(4)-H(4)	118.6(11)	C(15)-C(16)-H(16)	117.2(11)
C(4)-C(5)-C(6)	119.89(19)	C(18)-C(17)-C(16)	119.65(18)
C(4)-C(5)-H(5)	118.1(11)	C(18)-C(17)-H(17)	122.0(11)
C(6)-C(5)-H(5)	121.9(11)	C(16)-C(17)-H(17)	118.4(11)
C(5)-C(6)-C(7)	121.16(18)	C(17)-C(18)-C(19)	121.84(18)
C(5)-C(6)-H(6)	118.4(9)	C(17)-C(18)-H(18)	119.5(10)
C(7)-C(6)-H(6)	120.5(9)	C(19)-C(18)-H(18)	118.6(10)
C(6)-C(7)-C(2)	117.98(16)	C(18)-C(19)-C(14)	117.58(16)
C(6)-C(7)-C(11)	118.56(16)	C(18)-C(19)-C(23)	119.10(16)
C(2)-C(7)-C(11)	123.36(16)	C(14)-C(19)-C(23)	123.29(15)
C(3)-C(8)-C(9)	113.93(16)	C(15)-C(20)-C(21)	113.64(16)
C(3)-C(8)-C(10)	109.79(15)	C(15)-C(20)-C(22)	111.07(16)
C(9)-C(8)-C(10)	109.79(16)	C(21)-C(20)-C(22)	111.15(18)
C(3)-C(8)-H(8)	109.1(9)	C(15)-C(20)-H(20)	105.7(9)
C(9)-C(8)-H(8)	107.3(9)	C(21)-C(20)-H(20)	107.8(9)
C(10)-C(8)-H(8)	106.7(9)	C(22)-C(20)-H(20)	107.1(9)
C(8)-C(9)-H(9A)	113.3(12)	C(20)-C(21)-H(21A)	113.2(11)
C(8)-C(9)-H(9B)	111.9(11)	C(20)-C(21)-H(21B)	111.1(11)
H(9A)-C(9)-H(9B)	107.2(15)	H(21A)-C(21)-H(21B)	103.2(15)
C(8)-C(9)-H(9C)	109.9(10)	C(20)-C(21)-H(21C)	111.1(11)
H(9A)-C(9)-H(9C)	107.9(15)	H(21A)-C(21)-H(21C)	106.1(16)
H(9B)-C(9)-H(9C)	106.4(14)	H(21B)-C(21)-H(21C)	111.8(16)
C(8)-C(10)-H(10A)	111.0(11)	C(20)-C(22)-H(22A)	112.1(11)
C(8)-C(10)-H(10B)	111.6(11)	C(20)-C(22)-H(22B)	110.3(11)
H(10A)-C(10)-H(10B)	104.6(15)	H(22A)-C(22)-H(22B)	107.2(15)
C(8)-C(10)-H(10C)	111.0(10)	C(20)-C(22)-H(22C)	110.4(11)
H(10A)-C(10)-H(10C)	110.0(15)	H(22A)-C(22)-H(22C)	110.2(15)
H(10B)-C(10)-H(10C)	108.4(15)	H(22B)-C(22)-H(22C)	106.5(15)
C(12)-C(11)-C(7)	112.33(17)	C(19)-C(23)-C(25)	113.15(16)
C(12)-C(11)-C(13)	110.11(18)	C(19)-C(23)-C(24)	110.31(17)
C(7)-C(11)-C(13)	111.27(16)	C(25)-C(23)-C(24)	110.99(19)
C(12)-C(11)-H(11)	105.7(9)	C(19)-C(23)-H(23)	108.2(10)
C(7)-C(11)-H(11)	109.2(9)	C(25)-C(23)-H(23)	105.3(10)
C(13)-C(11)-H(11)	107.9(9)	C(24)-C(23)-H(23)	108.6(10)
C(11)-C(12)-H(12A)	113.6(11)	C(23)-C(24)-H(24A)	109.1(12)
C(11)-C(12)-H(12B)	111.4(11)	C(23)-C(24)-H(24B)	111.0(11)
H(12A)-C(12)-H(12B)	106.6(15)	H(24A)-C(24)-H(24B)	109.1(16)
C(11)-C(12)-H(12C)	109.4(12)	C(23)-C(24)-H(24C)	110.8(11)
H(12A)-C(12)-H(12C)	106.9(16)	H(24A)-C(24)-H(24C)	110.4(17)
H(12B)-C(12)-H(12C)	108.9(16)	H(24B)-C(24)-H(24C)	106.4(16)
C(11)-C(13)-H(13A)	110.6(11)	C(23)-C(25)-H(25A)	111.5(11)
C(11)-C(13)-H(13B)	110.7(10)	C(23)-C(25)-H(25B)	112.1(12)
H(13A)-C(13)-H(13B)	106.3(15)	H(25A)-C(25)-H(25B)	106.7(16)
C(11)-C(13)-H(13C)	109.5(12)	C(23)-C(25)-H(25C)	112.1(12)
H(13A)-C(13)-H(13C)	110.6(16)	H(25A)-C(25)-H(25C)	108.6(16)
H(13B)-C(13)-H(13C)	109.1(16)	H(25B)-C(25)-H(25C)	105.5(16)
C(19)-C(14)-C(15)	121.65(16)	N(3)-C(26)-C(28)	112.37(15)

N(3)-C(26)-C(27)	111.26(15)
C(28)-C(26)-C(27)	112.26(17)
N(3)-C(26)-H(26)	103.0(8)
C(28)-C(26)-H(26)	108.7(8)
C(27)-C(26)-H(26)	108.8(9)
C(26)-C(27)-H(27A)	110.1(11)
C(26)-C(27)-H(27B)	109.1(11)
H(27A)-C(27)-H(27B)	113.1(15)
C(26)-C(27)-H(27C)	111.6(10)
H(27A)-C(27)-H(27C)	103.2(15)
H(27B)-C(27)-H(27C)	109.6(15)
C(26)-C(28)-H(28A)	109.3(10)
C(26)-C(28)-H(28B)	111.0(12)
H(28A)-C(28)-H(28B)	111.0(16)
C(26)-C(28)-H(28C)	113.1(11)
H(28A)-C(28)-H(28C)	103.0(14)
H(28B)-C(28)-H(28C)	109.2(15)
N(3)-C(29)-C(31)	110.83(15)
N(3)-C(29)-C(30)	112.09(15)
C(31)-C(29)-C(30)	111.30(16)
N(3)-C(29)-H(29)	96.7(9)
C(31)-C(29)-H(29)	110.9(9)
C(30)-C(29)-H(29)	114.2(9)
C(29)-C(30)-H(30A)	112.0(10)
C(29)-C(30)-H(30B)	107.8(10)
H(30A)-C(30)-H(30B)	110.2(14)
C(29)-C(30)-H(30C)	110.8(11)
H(30A)-C(30)-H(30C)	111.0(14)
H(30B)-C(30)-H(30C)	104.8(15)
C(29)-C(31)-H(31A)	110.4(9)
C(29)-C(31)-H(31B)	110.6(11)
H(31A)-C(31)-H(31B)	108.6(14)
C(29)-C(31)-H(31C)	109.6(11)
H(31A)-C(31)-H(31C)	108.1(14)
H(31B)-C(31)-H(31C)	109.4(15)

Table 4. Anisotropic displacement parameters ($Å^2 x 10^4$) for 5. The anisotropic displacement factor exponent takes the form: $-2\pi^2 [h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
P(1)	187(2)	186(3)	193(3)	-4(2)	9(2)	0(2)
N(1)	149(7)	158(8)	159(8)	-7(7)	-4(6)	5(6)
N(2)	142(8)	154(8)	174(8)	-21(6)	-1(6)	1(6)
N(3)	160(8)	173(8)	192(8)	6(6)	-2(6)	-16(6)
C(1)	218(10)	198(10)	189(10)	1(8)	-2(8)	8(8)
C(2)	125(9)	200(10)	182(10)	-40(8)	-11(7)	18(7)
C(3)	125(9)	222(10)	195(10)	-18(8)	-14(7)	6(7)
C(4)	256(11)	313(12)	185(11)	4(9)	56(9)	31(9)
C(5)	330(12)	269(11)	275(12)	-83(10)	75(9)	69(9)
C(6)	285(11)	202(11)	304(12)	-7(9)	50(9)	50(9)
C(7)	161(9)	208(10)	217(10)	-13(8)	8(8)	16(8)
C(8)	161(10)	258(11)	193(10)	8(8)	39(8)	24(8)
C(9)	296(13)	339(13)	260(12)	40(10)	26(10)	48(11)
C(10)	276(12)	294(13)	318(13)	53(11)	21(10)	-55(10)
C(11)	294(11)	182(10)	225(11)	-9(9)	55(9)	64(9)
C(12)	344(14)	408(15)	312(14)	35(12)	79(11)	-53(11)
C(13)	340(13)	470(16)	282(14)	131(12)	-27(10)	-35(12)
C(14)	123(9)	208(10)	174(10)	-36(8)	14(7)	-18(7)
C(15)	164(9)	188(10)	201(10)	-10(8)	-4(8)	-4(8)
C(16)	267(11)	195(11)	273(11)	-45(9)	-15(9)	23(9)
C(17)	282(11)	255(11)	205(11)	-76(9)	-5(9)	-1(9)
C(18)	238(11)	281(12)	165(10)	26(9)	-14(8)	7(9)
C(19)	168(9)	199(10)	175(10)	-9(8)	12(7)	-18(8)
C(20)	253(11)	179(10)	235(11)	-23(9)	-76(9)	-4(9)
C(21)	403(14)	280(13)	329(14)	-15(11)	-89(12)	110(11)
C(22)	365(14)	399(14)	280(13)	117(11)	-24(11)	52(12)
C(23)	323(11)	228(10)	158(10)	-10(9)	-46(8)	6(9)
C(24)	484(17)	285(14)	506(17)	78(12)	62(13)	-89(11)
C(25)	356(14)	265(13)	463(16)	13(11)	-49(11)	83(10)
C(26)	178(10)	253(11)	252(11)	4(9)	9(8)	-35(8)
C(27)	247(12)	332(13)	327(13)	-6(11)	90(10)	-3(10)
C(28)	313(13)	293(12)	398(14)	-104(11)	96(11)	-104(10)
C(29)	199(10)	217(10)	222(10)	31(8)	-38(8)	-2(8)
C(30)	208(11)	211(11)	350(13)	29(10)	-37(10)	7(9)
C(31)	245(12)	298(12)	233(12)	14(10)	-33(9)	-12(10)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for 5.

	x	y	z	U_{iso}
H(4)	5709(17)	1933(6)	11081(14)	26(5)
H(5)	5329(19)	2741(7)	10533(14)	29(5)
H(6)	5773(16)	2994(6)	8823(12)	13(4)
H(8)	7330(17)	940(6)	9760(12)	15(4)
H(9A)	7840(20)	1241(7)	11534(15)	37(6)
H(9B)	7192(18)	699(7)	11500(14)	33(6)
H(9C)	6130(20)	1169(6)	11722(14)	32(5)
H(10A)	4260(20)	982(7)	10188(15)	41(6)
H(10B)	5157(19)	482(7)	10075(14)	35(6)
H(10C)	4893(17)	821(6)	9075(15)	25(5)
H(11)	7016(16)	2173(6)	6814(12)	15(4)
H(12A)	7470(20)	3201(7)	7459(15)	39(6)
H(12B)	7899(19)	2965(7)	6373(15)	33(5)
H(12C)	8660(20)	2785(7)	7449(15)	45(7)
H(13A)	4680(20)	2911(8)	7052(16)	55(7)
H(13B)	4380(20)	2316(7)	6682(14)	40(6)
H(13C)	5250(20)	2690(7)	5965(16)	36(6)
H(16)	7483(19)	-581(7)	5101(14)	34(5)
H(17)	6453(18)	-203(7)	3550(14)	31(5)
H(18)	5988(17)	671(6)	3484(13)	19(5)
H(20)	8888(16)	59(6)	7325(12)	9(4)
H(21A)	8740(20)	-930(8)	6475(15)	36(6)
H(21B)	9740(20)	-773(7)	7455(16)	37(6)
H(21C)	10030(20)	-556(7)	6318(15)	38(6)
H(22A)	6470(20)	-36(7)	7833(14)	36(6)
H(22B)	7520(20)	-449(7)	8381(16)	43(6)
H(22C)	6440(20)	-612(7)	7358(15)	45(6)
H(23)	6548(17)	1521(6)	5593(13)	21(5)
H(24A)	8670(30)	1547(8)	4602(16)	55(7)
H(24B)	7530(20)	2008(8)	4283(15)	44(6)
H(24C)	7520(20)	1532(7)	3537(16)	40(6)
H(25A)	4750(20)	1406(8)	3693(17)	50(6)
H(25B)	4720(20)	1872(8)	4483(15)	47(6)
H(25C)	4150(20)	1360(7)	4864(15)	45(6)
H(26)	12479(17)	1214(6)	8478(13)	19(5)
H(27A)	12205(19)	973(8)	6619(14)	37(6)
H(27B)	13380(20)	1441(7)	6834(14)	44(6)
H(27C)	11730(20)	1514(7)	6275(15)	35(6)
H(28A)	11120(20)	2158(7)	7716(14)	27(5)
H(28B)	12800(20)	2109(7)	8261(15)	48(6)
H(28C)	11340(20)	2034(7)	8929(15)	42(6)
H(29)	9020(19)	693(6)	8698(13)	30(5)
H(30A)	11208(17)	204(6)	8082(14)	22(5)
H(30B)	11048(19)	92(7)	9321(15)	39(6)
H(30C)	12220(20)	449(6)	8976(13)	25(5)
H(31A)	11427(18)	1178(6)	10063(12)	15(4)
H(31B)	10244(19)	811(7)	10522(15)	41(6)
H(31C)	9730(20)	1343(8)	9981(14)	43(6)